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Implications of anionic and natural surfactants to measure wettability alteration in EOR processes

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Abstract

Minerals surface properties have played a substantial to predict the rock mineralogy and chemical materials interactions, especially in chemical flooding (e.g., polymers, surfactant). In this paper, two different surfactants; nonionic surfactant (Zizyphus Spina Christi) and anionic surfactant (henceforth; SDBS) were used to experimentally investigate the minerals of reservoir rocks especially carbonate reservoirs and measure wettability changes accordingly. The contact angle evaluations have depicted that utilized surfactants change the wettability of pellet surfaces of calcite, dolomite, quartz, and anhydrite to neutral-wet or slightly water-wet. As it was observed, SDBS provided the maximum wettability changes for quartz surface. Moreover, the Zizyphus Spina Christi and SDBS have provided efficient performances on the decrease of wettability changes and residual oil saturation in dolomite core and quartz core, respectively. According to the results of this study, the oil recovery factor with anionic SDBS surfactant for calcite, dolomite, and quartz plugs are 66%, 41%, and 93%, respectively. This increase is more visualized for quartz cores that indicated the compatibility of this surfactant with quartz core samples.

Keywords: Wettability Change; Zizyphus Spina Christi; Contact angle; SDBS; Enhanced Oil Recovery

Abbreviation

Sor	Residual oil saturation
EOR	Enhance oil recovery
COBR	Crude oil/brine/rock system
SDBS	Sodium Dodecyl Benzene Sulfonate
Swi	Initial water saturation

1. Introduction

Oil production has been intentionally declined gradually, and it results in the consequential oil crisis, such as boosting the oil price. Approximately two-thirds of the original oil in place in a reservoir is still maintained after primary and secondary recovery stages, and efficient EOR methods are needed for more oil production (Lake et al., 2014; Davarpanah and Mirshekari, 2020; Sheng, 2010; Alzahid et al., 2019). Chemical injection methods are mostly known for the way of producing residual oil after water flooding. These methods have been used for decreasing the interfacial tension, raising the brine viscosity as a mobility control, and increasing sweep efficiency in tertiary recovery (Taber et al., 1997; Shandrygin and Lutfullin, 2008; Rai et al., 2015; Davarpanah, 2018; Mejia et al., 2019). The wettability of an oil reservoir affects the fluids flow displacement and its location in the porous media during the production, and it is a crucial parameter in oil recovery processes (Anderson, 1986; Zhang et al., 2012; Bahaloo Horeh et al., 2019; Emadi et al., 2019; Mofrad and Saeedi Dehaghani, 2020; Davarpanah and Mirshekari, 2019). The reservoir rock mineralogy has a significant impact in the interaction mechanism between externally added surfactants and reservoir minerals and their influences on solid-liquid interfacial properties such as wettability and surface charge

(Somasundaran and Zhang, 2006; Tu and Sheng, 2020; Chávez-Miyauchi et al., 2016). There is various rocks mineralogy that could alter the wettability, when the chemical agents have been injected into oil reservoirs (Zendehboudi et al., 2013; Peters and Faulder, 2012; Davarpanah et al., 2019; Emadi et al., 2017).

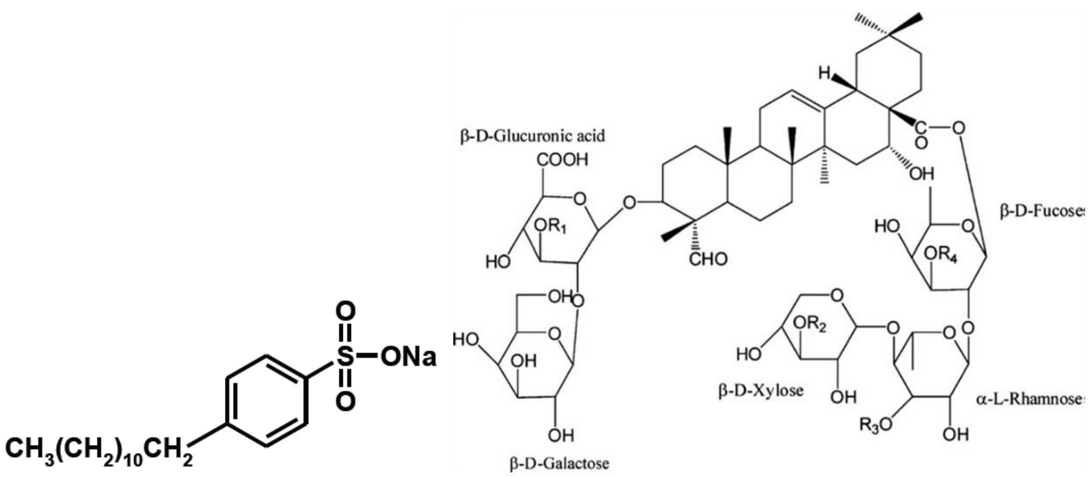
Chon et al. (2014) worked on the impact of anionic surfactant (dodecyl alkyl sulfate) for EOR process in sandstone reservoirs. This surfactant resulted in betterment in oil recovery from 23.9% (at 0 wt% surfactant) to 65.4% (at 2 wt% surfactant) with the crude oil(Ko et al., 2014). Bennetzen et al. (2014) probed the oil production from oil-wet limestone rock of the Al Shaheen field, using several surfactants. These results showed that 14.7-28% of OOIP was recovered using various surfactants(Bennetzen et al., 2014). Shadizadeh and Ahmadi (2013) implemented a novel sugar-based surfactant (Zyziphus Spina Christi) for enhanced oil recovery from carbonate reservoirs. In this core experiments, oil recovery enhanced from 55.45% (at 0 wt% surfactant) to 81.08% (at 8 wt% surfactant) with the Naft-Shahr crude oil(Ahmadi and Shadizadeh, 2013b)**.** Chen et al. (2013) figured out the new alkaline/surfactant solution with an alkyl polyglucoside for enhancing the substantial oil recovery in the different sand pack. These outcomes several that the tertiary oil recovery for several sand pack reach from 16% to 19% approximately(Chen et al., 2013). Anna Zdziennicka, Bronisław Jan´ czuk investigated wettability of quartz employing alcohol anionic surfactant. The investigation of the contact angle data points reveals that the changing wettability of quartz altered vividly only in the range of alcohol and anionic surfactant concentration (Zdziennicka and Jańczuk, 2011). Nermin Gence evaluated wettability of magnesite and dolomite surfaces and observed they had got a small contact angle without using any surfactant. If there are petroleum sulphonate (R825 and R840) with sodium oleate, the contact angle value increased(Gence, 2006; Golabi et al., 2012).

Since the compositions of rock have the most influence on wettability and oil mobility of reservoir, recognizing the minerals in the reservoir and their behavior adjacent crude oil/brine/rock (COBR) system is an individual parameter during EOR progression. This research aims to study the performance of surfactant on the wettability and the oil recovery for various rocks mineralogy by operating different experimental measurement. The main reasons to opt these surfactants are. First, they are representing the two main groups of surfactants, and it has been proved that they are impressive on oil recovery. Secondly, the Cedar could be obtained from lots of Zizyphus Spina Christi trees by low cost in Iran, and it is environmentally friendly. Hence it can be utilized in EOR methods, mostly in chemical flooding regarding the Iranian carbonate reservoirs. Therefore, in this work, two surfactants including nonionic surfactant (Zizyphus Spina Christi) and anionic surfactant (SDBS) were employed to elucidate the impact of wettability alteration on minerals of various reservoirs especially carbonate reservoir rocks using contact angle measurements, and the oil recovery of different plugs was measured by surfactant flooding.

2. Materials and Methods

2.1. Materials

Chemical Formula; two different surfactants that were used in this experiment are; nonionic surfactant (Zizyphus Spina Christi was easily extracted with low cost from trees, and it is not harmful to the safety environment) and anionic surfactant (SDBS is sodium dodecylbenzene sulfonate (CH_3 $(CH_2)_{11}C_6H_4SO_3Na$)). The chemical structure of these surfactants are schematically depicted in Figure 1 (Golabi et al., 2012; Stanimirova et al., 2011). Ball milling is one of the non-equilibrium technique to produce strengthened alloys that are contained fine microstructures. This process contained mechanical alloying and mechanical milling to refine the size of grains and solid elements to the nanoscale. We use a ball milling method to refine grain sizes to nanoscale sizes in this study(Ullah et al., 2014).



A B

Figure 1.A) SDBS(Golabi et al., 2012) and B) Zizyphus Spina Christi(Ahmadi and Shadizadeh, 2013a)

Crude oil; the crude oil utilized in this work, was provided from Bangestan reservoir from Southern part of Iran. The viscosity and density of crude oil at ambient temperature are 26 cp and 0.88 g/cm^3. Crude oil composition is statistically explained in Table 1

Table 1. Crude oil Composition

Composition	**Mole%**	**Composition**	**Mole%**
C_1	20.14	C_8	3.42
C_2	6.59	C_9	4.13
C_3	5.14	C_{10}	3.84
nC_4	1.25	C_{11}	3.85
iC_4	2.96	C_{12+}	2.4
nC_5	1.54	CO_2	0.84
iC_5	2.36	H_2S	0
C_6	4.52	N_2	0.3
C_7	20.14		

Brine; Distilled water was utilized as the aqueous phase for all tests, because of the precipitation of surfactant when using brine (Deymeh et al., 2012). Brine properties are described in Table 2.

Table2. Formation brine components

Brine Type	TDS (mg/L)	pH (25 ℃)	pH (85 ℃)	Density (25 ℃) g/cm^3	Density (85 ℃) g/cm^3
KCl	1540	6.6-6.9	6.5-6.8	1-1.0045	0.985-0.99
$MgCl_2$	5142	6.8-7.1	6.65-7	0.95-1	0.98-0.985
$CaCl_2$	3125	6.7-7.1	6.5-7	1.0002-1.003	0.98-0.985
NaCl	112540	6.21-6.68	6.12-6.53	1-1.0025	0.975-0.98

Core Plugs; three plugs consist of calcite, dolomite, and quartz were used in this work which is shown in Figure 2. Two plugs were obtained from Chenare mine in the north of Khuzestan in Andimeshk city of Iran. One core plug is dolomite rock, and another is calcite rock. The reason for chosen these rocks is the purity amount of calcite and dolomite. Three thin sections were made from each core plug to obtain the percent of the existing minerals in these plugs. With a microscope, each of them was investigated, and minerals percent of each plug were determined. After all, by getting the average of them, the percent of existents minerals in each plug were estimated. This method is so accurate and practical in Petroleum Geology to determine the percentage of rock minerals. Besides, the Chenare mine is well known because of its calcite and dolomite pure.

Another plug used in this experimental work is quartz packed plug. Because the goal is the observation of the influence of mineralogy on oil recovery and the core plug with a high purity of quartz has not been found. There is not a sandstone reservoir with high quartz mineral purity, and therefore, the quartz pack was prepared from quartz crystalline. A 13.3 cm long cylindrical high pressure and temperature stainless steel were utilized as a particle-pack catcher. Pressure and temperature's constraints for this system are 4000 psi and 300 ^{o}F, in sequence. The high purity crystalline quartz was crushed and powdered and then passed through sieves of 45 and 50 mesh sizes. Rock very small fragments of 350-450 micron diameter were obtained after sieving. To hinder the exit of the fine quartz rock particles as during flooding, two filters were embedded on the inlet and outlet of the cylinder (Figure 3). The mesh size of the filter is approximately 300micron, which is smaller than the particles size. For the appropriate distribution of fluids and conservation of filters, the cylinder is equipped with two distributor plates (Mousavi et al., 2007). After the obtained rock particles with 350-450 micron, mixed approximately 92% quartz with 4% sandstone rock and 4% Portland cement. In order to strengthen and consolidation of this composition and prevention of sand migration, Portland cement was used.

The properties of the petrophysical and mineralogical data are presented in Tables3 and 4.

Figure 2. Core plugs consist of calcite, dolomite, and quartz

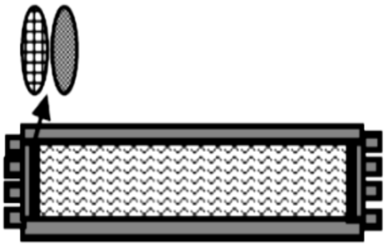


Figure 3. Schematic of the packed bed.

Table 3. The petrophysical properties of plugs

Core	L [cm]	D [cm]	Vb[cm3]	Φ [%]	K [mD]	PV [ml]
Calcite	12.2	3.8	138.3	4.7	6.02	6.5

Dolomite	14.4	3.8	163.3	8.2	24.5	13.4
Quartz	13.3	3.8	150.7	29.4	570	44.2

Table 4. The mineralogical composition of plugs

Core	Calcite(%wt)	Dolomite (%wt)	Quartz (%wt)	Clay (%wt)
Calcite	~90	~5	_	~5
Dolomite	~6-7	~85	~1-2	~6-7
Quartz	4	_	92	4

Pellet samples used in this study, are high purity crystalline of minerals; calcite, dolomite, quartz, gypsum, anhydrite, and shale. It is schematically depicted in Figure 4.

Figure 4. Pellet samples used in this study

2.2. Methods

- Contact angle evaluations

In the first step, polished and cleaned crystalline pellets (1cm in diameter and 4mm in thickness) were placed in the Soxhlet extractor to clean with toluene as a solvent. The prepared pellet samples were submerged in the crude oil and were placed in an oven at high temperature (around 60 ^{o}C) for at least 100 hours in order to become oil-wet(Peters, 2012). The contact angle measurement was evaluated in a three-phase system (Buckley and Liu, 1998). Captive

Drop instrument (made in Alberta Research Council, Canada, 2002) was used to measure wettability in this experiment. The various concentrations of surfactant were mixed in distilled water, and this homogenous solution was injected into the cell, and oil was suspended from the surface of the sample by a capillary tube. This procedure must be accomplished very masterfully to ensure that the oil drop is stabled to the sample surface (Zhang et al., 2012). The measurements were conducted at 25^{o}C, and images of the drop shape were captured with a digital camera (Figure 5).

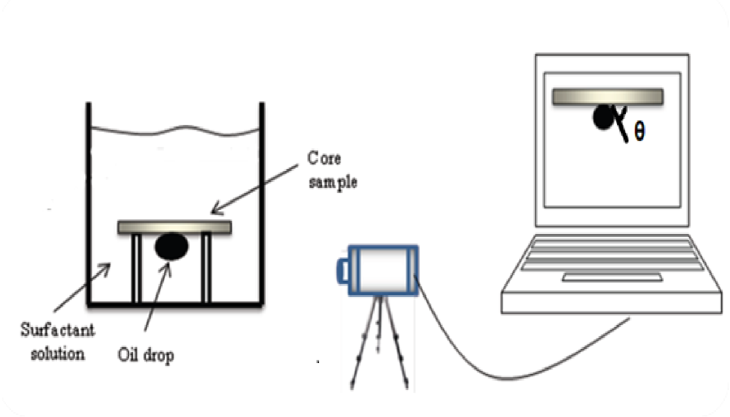


Figure 5. Schematic plan of the contact angle measurement.

- *Core flooding*

Figure 6 reveals a schematic diagram of the laboratory instrument for core flooding experiments. Fluid (brine, oil, and surfactant) is injected to the core plug by an HPLC pump. An overburden was sustained at 20 bars. Due to measuring the pressure drop across this process, a differential pressure transmitter was embedded to the inlet and outlet ends of the core, and the data were written down into the computer over and over. All the tests were implemented under experimental (pressure and temperature) conditions.

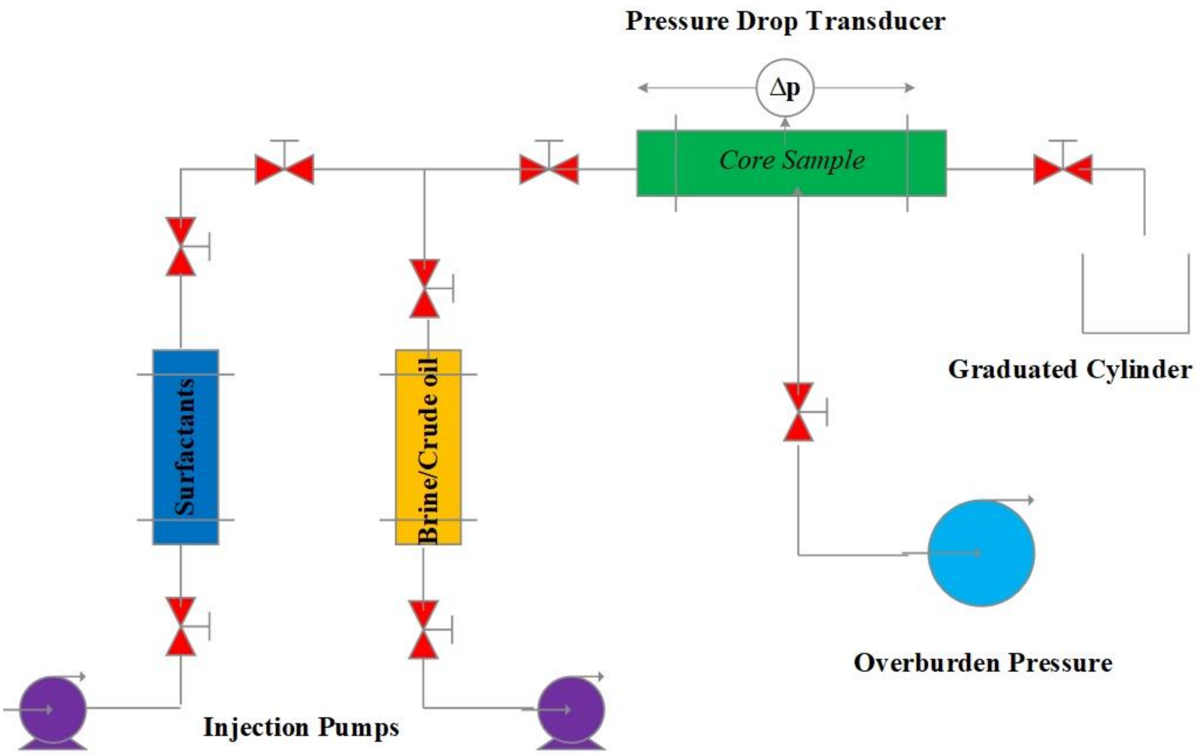


Figure 6. Schematic of core flooding experimental setup.

The steps of this procedure are as follows:

- The absolute permeability and porosity were obtained.
- The core plug was saturated with distilled water.
- Oil was injected at a low rate of 0.5 or 1 cc/min for 2 pore volumes because it should displace water with a uniform front.
- Then oil is injected with the rate of 2 or 3 cc/min to ensure that no more moveable water exists in plugs.
- In this stage, connate water saturation was in the plug, and distilled water cannot be detected in the effluent produced by the 1 or 2 pore volume of oil injection.
- At the next stage, water was injected at a low rate to determine the residual oil saturation (S_{or}). At first, the water was injected and then when oil had not been observed in the effluent, the produced oil was collected, and measured and residual oil saturation was obtained.

In this study, sleeve and core holder were used for coating and locating the plug, respectively. Overburden pressure was fixed, and distilled water began to inject from transfer vessel to plug with a constant flow rate. The pressure was recorded utilizing a pressure gauge. The oil recovery was calculated by measuring the oil production. Water flooding with distilled water was fulfilled after oil flooding to measurement the S_{or}. After injection of approximately 3 PV of distilled water into the core at a low constant flow rate of 0.2–0.5 cc/min, the flowing out fluids were produced, and the S_{or} was estimated. After measured these recovery processes, core samples are flooded with a surfactant solution. At this level, the surfactant solution injected to the core plugs, and the amount of oil production is measured for obtaining both enhanced oil recovery (EOR) and critical oil saturation.

3. Results and Discussion

3.1. Contact angle results

The impact of surfactants (SDBS and Zizyphus Spina Christi) on pellets of crystalline minerals wettability was performed by estimating the contact angle. The contact angle was measured between the surface pellet and oil drop-in (COBR) system (within about1–2 min after fixing the drop). Initially, the contact angle measurements (θ) for crystalline mineral pellets exposed to distilled water were: calcite 149°, dolomite 158°, quartz 142°, anhydrite 144°, gypsum 78° and shale 130°. These results indicated that the calcite, dolomite, quartz, anhydrite and shale were firmly oil-wet and gypsum was slightly water-wet.

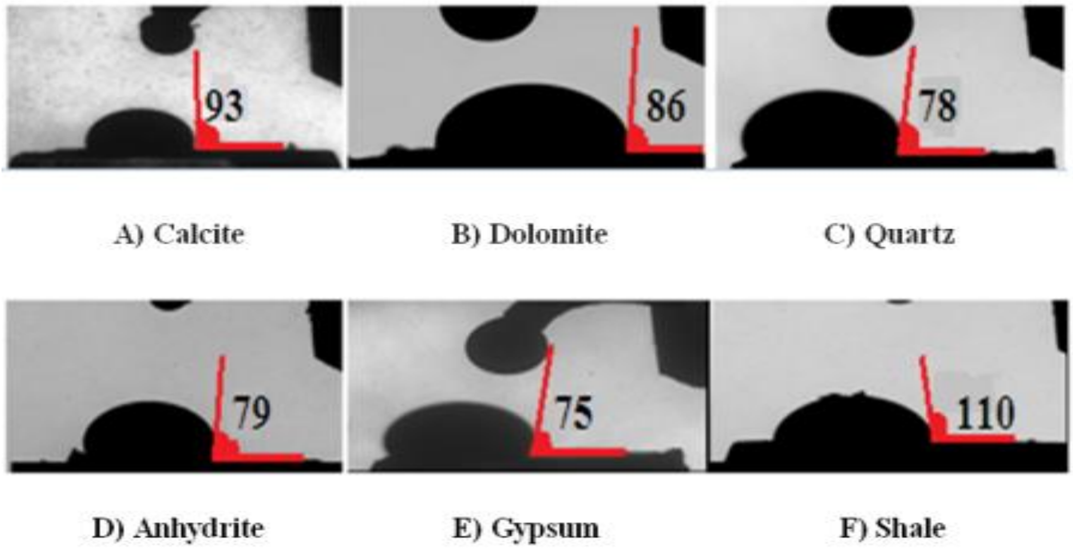


Figure 7. The contact angle of minerals for treated crystalline minerals exposed SDBS.

Anionic surfactant (SDBS) with various concentrations (0.05, 0.1, 0.2, 0.5, 1, 1.5, and 2 wt %) was used. The results for the aged calcite surface, treated at low concentration (0.05 wt%), show the contact angle changed from 149° to 140°; however, when the concentration was increased to the high level (2 wt%), the contact angle changed to 93°, and wettability of the calcite surface changed to neutral-wet. For the aged dolomite surface by changing the concentration (from 0.05 wt% to 2 wt %), the contact angle altered from 158° to 86°. For the aged quartz, anhydrite, gypsum, and the shale surfaces, the contact angle alteration was observed from 142° to 78°, 144° to 79°, 78° to 75° and 130° to 110°, respectively as shown in Figure 7. Figures 8 shows the contact angle with various concentrations of SDBS surfactant. Calcite, dolomite, quartz and anhydrite surfaces have almost the same behavior exposed to SDBS, but this surfactant is not enough effective on gypsum and shale surfaces wettability alteration. It is plotted in Figure 8.

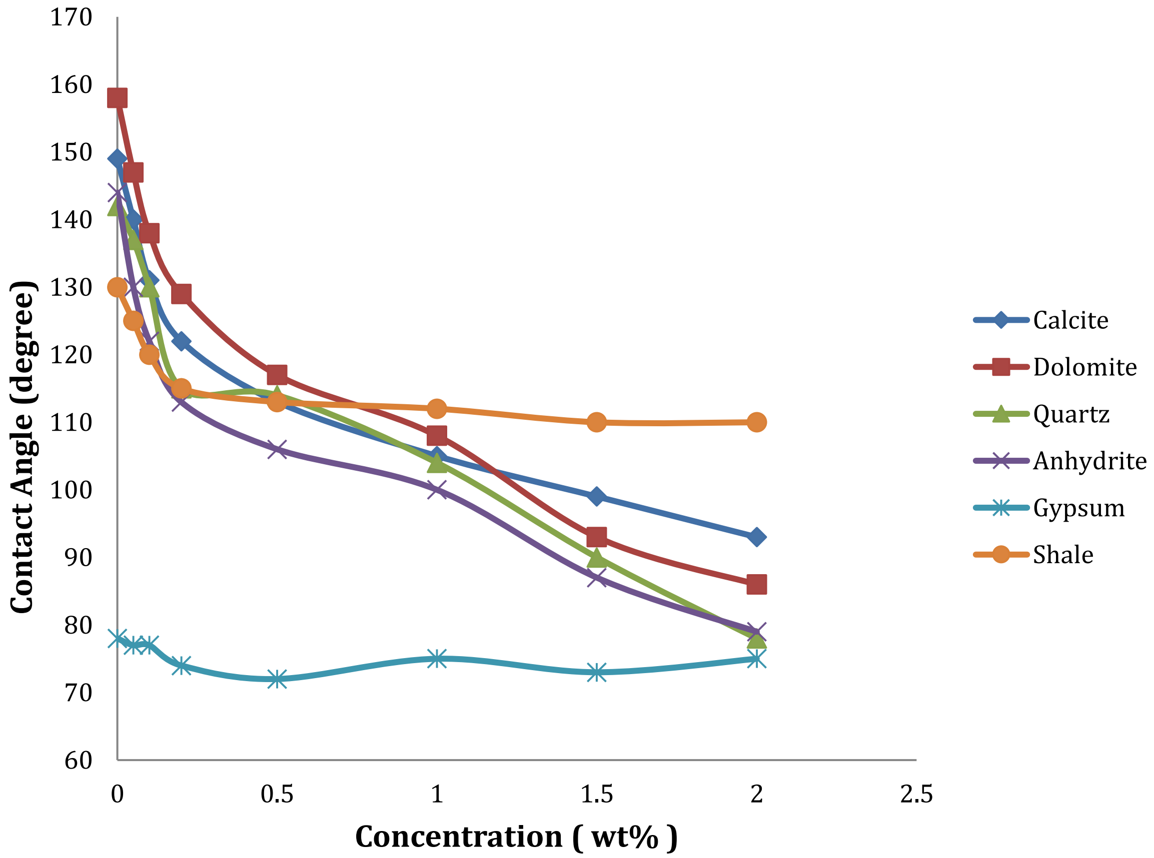


Figure 8. Effect of SDBS concentration on the contact angle.

The effect of various concentrations (0.5, 1, 1.5, 2, 3, 4, and 6 wt %) of nonionic surfactant (Zizyphus Spina Christi) on wettability alteration is shown in Figure 9. For the aged calcite surface, treated at low concentration (0.5 wt%), the results illustrate that the contact angle altered from 149° to 144°, however when the concentration increased to the high level (6wt%), the contact angle changed to 110°, and wettability of the calcite surface changed to neutral-wet. For the aged dolomite surface, by changing concentration (from 0.5wt% to 6 wt %), the contact angle altered from 158° to 98°. For the quartz, anhydrite, gypsum and the shale surfaces, the contact angle altered from 142° to 90°, 144° to 89°, 78° to 72° and 130° to 119°, respectively. It is schematically depicted in Figure 9.

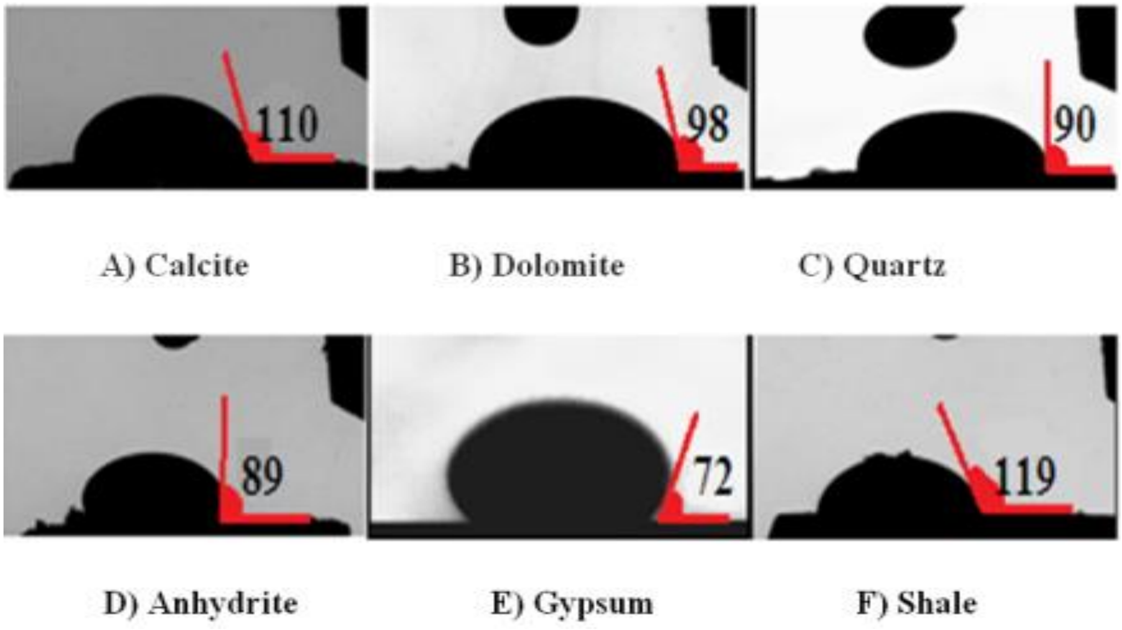


Figure 9. The contact angle of minerals for treated crystalline minerals exposed Zizyphus Spina Christi.

The effect of Zizyphus Spina Christi wettability alteration of mineral surfaces is less than SDBS. This surfactant changes the wettability of calcite and dolomite surfaces from strongly oil-wet to slightly oil-wet, and the wettability of quartz and anhydrite surfaces from strongly oil-wet toward neutral-wet. Zizyphus Spina Christi surfactant has no enough impact on wettability alteration of gypsum and shale surfaces. The performance of the used surfactants for the most reduction of contact angle and changed wettability alteration for calcite, dolomite, and anhydrite surfaces are in order SDBS and Zizyphus Spina Christi. For quartz surface, the most wettability alteration is obtained by SDBS and then Zizyphus Spina Christi, respectively. Nevertheless, these surfactants for shale and gypsum surfaces have no enough effect for reduction contact angle. These results confirm that this type of surfactants changes the wettability of surfaces of calcite, dolomite, quartz and anhydrite to neutral-wet or slightly water-wet and they did not change the wettability of gypsum and shale surfaces a lot. It is schematically depicted in Figure 10.

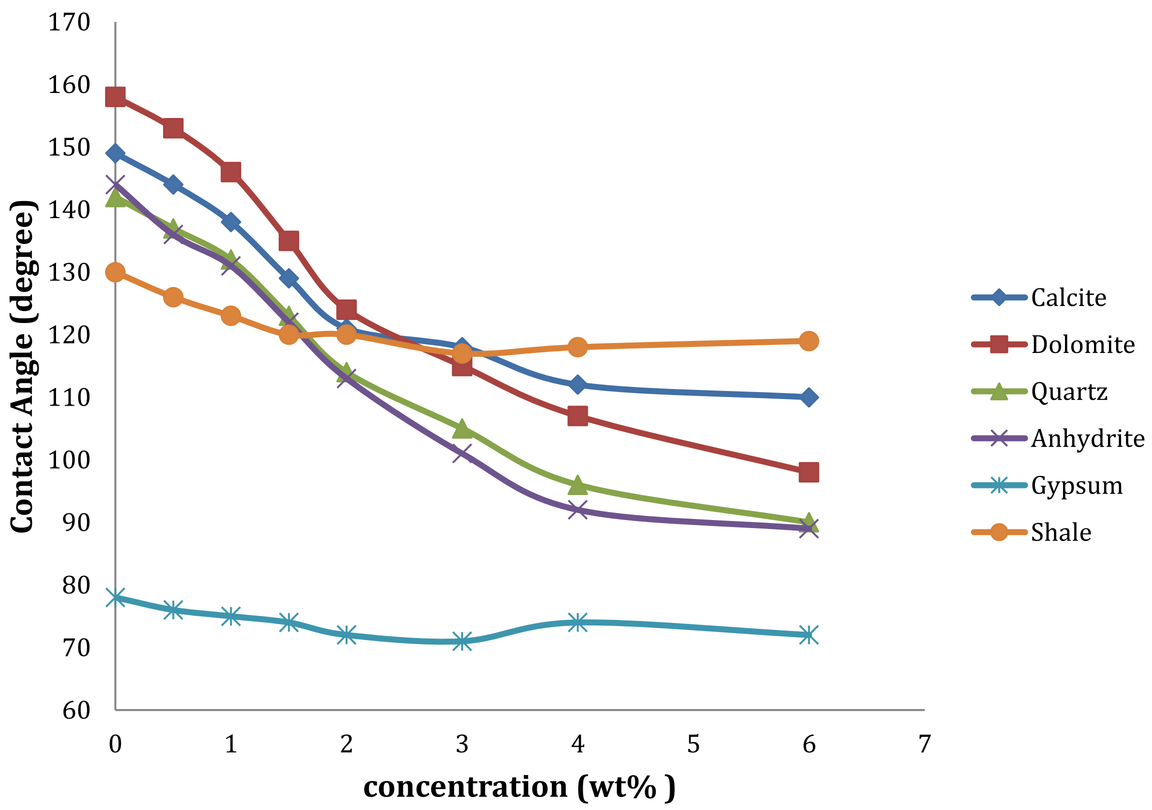


Figure 10. Effect of Zizyphus Spina Christi concentration on the contact angle.

3.2. Oil Saturation

As depicted in Table 3, the initial oil saturation and residual oil saturation for the calcite plug after water injection are 79% and 46%, respectively. Also, the related results for dolomite plug are 76%and 56%, reciprocally. Furthermore, finally, the measurements of quartz plug are 61%and 23%, individually. By water flooding, the residual oil saturation obtained for calcite, dolomite, and quartz plugs is 46%, 56%, and 23%. After the surfactant flooding with nonionic Zizyphus Spina Christi surfactant, the residual oil saturation for calcite plug is 36.3%, for dolomite plug is 25%, and for quartz, the plug is 8.5%. These results show that the most decrement in the S_{or} has occurred for dolomite plug, 31%, the second decrease in S_{or} has happened for quartz plug, 14.5%, and the lowest reduction in residual oil saturation related to calcite plug is 9.7%. These results confirm that the nonionic Zizyphus Spina Christiis effective in changing the wettability of calcite, dolomite, and quartz plugs. The most wettability alteration and oil recovery are obtained for dolomite, quartz, and calcite plugs, respectively. After surfactant flooding with anionic SDBS surfactant, the residual oil saturation for calcite plug is 26.8%, for dolomite plug is 44.8%, and for quartz, the plug is 4.3%. These results show the most reduction in residual oil saturation occurred in calcite plug, 19.2%, the second decline in residual oil saturation occurred in quartz plug, 18.7%, and relates the lowest decrease in residual oil saturation to dolomite plug, 11.2%. These results confirm that the anionic SDBS is efficient in altering the wettability of calcite, dolomite, and quartz plugs. The most wettability alteration and oil recovery are obtained for quartz, calcite, and dolomite plugs, respectively. It is shown statistically in Table 5.

Table 5. Variation of residual oil saturation during flooding.

	Calcite core	Dolomite core	Quartz core
Initial oil saturation	79%	76%	61%
Residual oil saturation by water flooding	46%	56%	23%
Residual oil saturation by Zizyphus Spina Christi flooding	36.3%	25%	8.5%
Residual oil saturation by SDBS flooding	26.8%	44.8%	4.3%

3.2. Recovery Factor

Figure 11 illustrates the oil recovery curves in different flooding processes. In water flooding, at first, oil recovery increases sharply up to breakthrough. In this period, oil is the only effluent fluid from the core sample. In the second region, the recovery graph is slightly changed or remains virtually unchanged. It shows that the most of the oil which is located in the larger pore spaces is depleted and the others which are distributed in the small pores require a high viscose force to overcome the capillary pressure in order to produce.

Core flooding experiments were fulfilled in three various minerals (calcite, dolomite, and quartz plugs). Two sorts of surfactants were employed: nonionic surfactant (Zizyphus Spina Christi and anionic surfactant (SDBS). Due to the outcomes of the contact angle tests, the optimum concentration of SDBS is around 1%wt, and the best concentration of Zizyphus Spina Christi is 4%wt. The investigations of oil recovery were conducted using 4%wt concentration of nonionic Zizyphus Spina Christi surfactant (according to the contact angle results) showed an improvement in recovery of oil from plugs. According to figure 11, the oil recovery with nonionic Zizyphus Spina Christi surfactant for calcite, dolomite, and quartz plugs are 54%, 67%, and 86%, respectively. Therefore the oil recovery for calcite plug increases from 41.7% to 54%, for dolomite plug increase from 26.3% to 67%, and for quartz, plug increases from 62.2 % to 86%. These results show the most increment of oil recovery related to dolomite plug, 40.7%, the mildest increment of oil recovery related to quartz plug, 23.8%, and the lowest increment oil recovery among these rocks related to calcite plug, 12.3%. The investigations of the oil recovery were conducted using 1%wt concentration of anionic SDBS surfactant (according to the contact angle results) showed an improvement in recovery of oil from plugs. As can be seen from this figure, the oil recovery with anionic SDBS surfactant for calcite, dolomite, and quartz plugs are 66%, 41%, and 93%, respectively. Therefore oil recovery for calcite plug increases from 41.7% to 66%, for dolomite plug increase from 26.3% to 41%, and the quartz plug increases 62.2 % to 93%. These results relate the most increment of oil recovery to quartz plug, 30.8%, the second increment of oil recovery to calcite plug, 24.3%, and the lowest increment oil recovery among these rocks to dolomite plug, 14.7%.

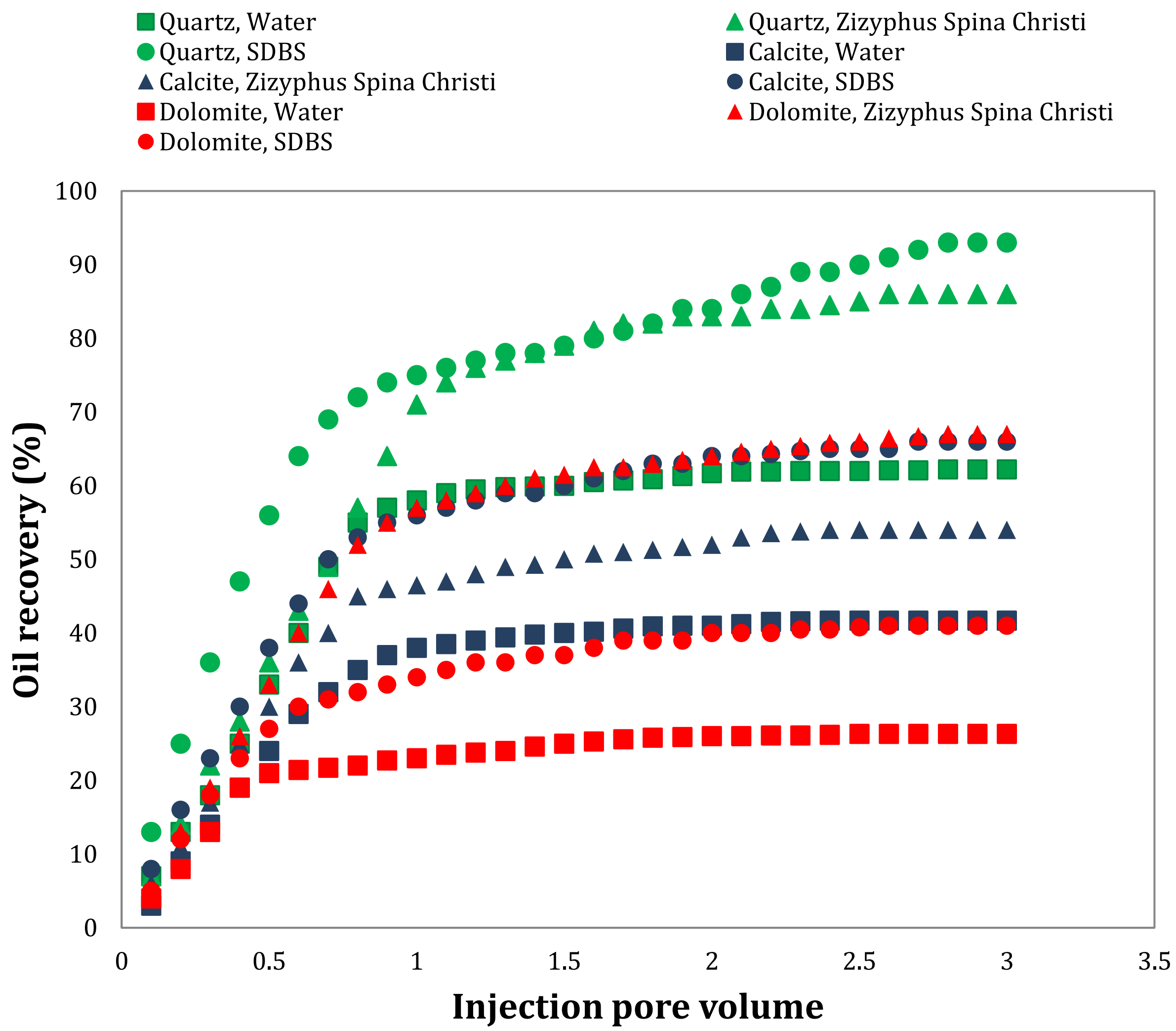


Figure 11. Oil recovery results of the flooding processes.

According to the obtained results and compare these outcomes with previous works, it can be concluded that these surfactants have a significant impact on the oil industry. For instance, some studies on natural anionic surfactant by Mandal et al. (2019), nonionic surfactant on sandstone minerals by Barati-Harooni et al. (2016) were done to consider these surfactants impact on the oil recovery factor (Saxena et al., 2019; Barati-Harooni et al., 2016). Further studies were done to investigate the effect of natural surfactants on carbonate minerals by Ahmadi and Shadizadeh (2013)(Ahmadi and Shadizadeh, 2013a). Additionally, in this research, we investigate the behavior of different types of minerals in touch with surfactant in reservoirs rocks and therefore, it gives the perfect view in wettability alteration and mineralogy dependency.

4. Conclusions

Surfactant flooding is considered as one of the efficient chemical enhanced oil recovery techniques in recent decades as it has the potential ability to alter the wettability and reduce the interfacial tension of water-oil. The main conclusions of this paper are as follows;

- ✓ Both surfactants are useful in decreasing contact angle, while SDBS is more effective than Zizyphus Spina Christi in altering the wettability of calcite, dolomite, and anhydrite surfaces from strongly oil-wet toward neutral-wet or slightly water-wet.
- ✓ For the quartz surface, the most wettability alteration is obtained by an anionic surfactant (SDBS). This wettability alteration is from strongly oil-wet to neutral-wet.
- ✓ About the calcite core, the anionic surfactant (SDBS) was more useful for changing the wettability and reduction of residual oil saturation and enhances oil recovery.
- ✓ For the quartz core, the anionic surfactant (SDBS) was more active in changing the wettability and enhances oil recovery.
- ✓ In the dolomite core, the nonionic surfactant (Zizyphus Spina Christi) was more useful on changing the wettability and decrease of S_{or}.
- ✓ The compositions of the rock have the most influence on wettability and oil mobility of reservoirs, therefore, recognizing the minerals in the reservoir and their behavior adjacent crude oil/brine/rock (COBR) system is a vital parameter in the processes of enhanced oil recovery (EOR).

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